ABSTRACT

New possibilities to obtain concentrates of PUFA esters by high vacuum distillation

NICOLETA-GABRIELA ŞTEFAN[1], PETRICA IANCU[1], VALENTIN PLEŞU[1*], AURELIAN TOMA[1] , ALEXANDRA –ELENA BONET-RUIZ[1] , JORDI BONET-RUIZ[2]

[1)] Centre for Technology Transfer for the Process Industries, University POLITEHNICA of Bucharest, 1, Gh. POLIZU Street, Building A, Room A056, RO-011061 BUCHAREST, ROMÂNIA

[2)] Department of Chemical Engineering, University of Barcelona, 1, Martí i Franquès Street, E-08028 Barcelona, Spain

New sustainable processes to produce enrich ω food supplements from *Camelina sativa* oil as feedstock is developed. ω compounds have large molecules and can be easily degraded if the separation is performed at atmospheric pressure. The new process involves the use of high vacuum fractionation and molecular distillation to obtain ω concentrates. In first of all, triglycerides from camelina oil are converted into fatty acid methyl esters because they are easier to separate due to their higher volatility. Then, high vacuum fractionation (up to 1 Pa) and molecular distillation (up to 0.1 Pa) are needed in order to avoid the degradation of ω fatty acid esters molecular structure. These techniques lower the boiling points and are an excellent method for gentle thermal treatment of heat sensitive products (Iancu et al., 2015).

Some data about vapor pressure of ω-fatty acids methyl esters indicate the potential for separation. Thus the vapor pressure, estimated from literature data (Batistella et al., 2002; Rossi et al., 2011), at 100°C for C18:2 methyl ester is 0.67 Pa, and for C18:3 methyl ester is 0.53 Pa, while the vapor pressure for C20:1 methyl ester is 0.31 Pa. Using same literature source (Batistella et al., 2002; Rossi et al., 2011), at 120°C the vapor pressure for C18:2 methyl ester is 4.30 Pa, and for C18:3 methyl ester is 3.50 Pa, while for C20:1 methyl ester is 1.70 Pa. In conclusion, the minimum pressure for distillation is 1 Pa. Even at high vacuum, the omega-3 fatty acid methyl ester (C18:3) will be very difficult to separate due to the low vapour pressure difference. C18:3 and C18:2 have very close boiling points.

Molecular distillation is a technique that involves vaporized compounds traveling a short distance, only a few centimeters, from an evaporator surface to a condenser surface. The operation is normally performed at reduced pressure, down to 0.1 Pa. A pressure gradient from evaporator to condenser is thereby essentially avoided, and vapor molecules may travel between the evaporator and the condenser without colliding with other molecules.

The fatty acid methyl esters mixture is fed into the high vacuum distillation plant, at preparative scale DSL5 from UIC GmbH. Esters get to evaporation unit where a liquid film is formed by the action of rollers attached to a wiper basket. From the thin film evaporator the esters go to the rectification column with Montz@ structured packing. In the rectification column, the compounds are separated according to the differences in volatility. The fraction of light esters (compounds with fewer carbon atoms) reaches the top of the rectification column and condensed, while the heavy ones (compounds with higher number of carbon atoms) go back to the bottom of the rectification column, and then into the thin film evaporator, where are collected as bottom product. The ratio between bottom product and distillate product is established at 1:1. The vapor temperature is settled between 164.2 - 167.1°C. In this temperature range, the required reflux ratio is stabilized. The pressure is settled between 2.8-3.5 Pa. Then, a second high vacuum fractionation step is performed. The bottom product from the first high vacuum distillation step is fed in the plant. The parameters are settled between 172.1-174.3°C and the pressure between 2.4- 3 Pa. The distillate resulted in the second high vacuum distillation step is separated by molecular distillation in a laboratory pilot plant, at preparative scale - KDL5 from UIC GmbH, Germany. The operation temperature is varied between 90-110°C to evaluate the relationship between temperature and efficiency of ω fatty acids esters separation. The system pressure is maintained constant (0.1 Pa).

Compared to the original mixture, containing ~47% ω fatty acids esters, their final concentration increased to ~74.4%.

References:

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